

# **Improved Crystal Quality by Detached Solidification in Microgravity**

**Final Report  
1 February 1998 to 31 January 2003**

**NASA Grant NAG8-1482**

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## Summary

The goals of our work on detached solidification have been to:

- Develop a complete understanding of all of the phenomena of detached solidification.
- Make it possible to achieve detached solidification reproducibly.
- Increase crystallographic perfection through detached solidification

Specifically, we aimed in this project to:

- Identify a system and develop methods that would allow viewing of the melt surface and convection in the melt during detached solidification in microgravity.
- Improve understanding of the origination and evolution of detachment through experiments and theoretical treatments.
- Achieve detachment on earth.

The project resulted in 14 publications, 15 presentations, completion of 2 Ph.D. theses, and completion of 2 M.S. theses. Two additional papers are currently being reviewed for publication. Copies of most of the papers are attached as appendices.

Among the accomplishments are:

- Achievement of detached solidification of InSb on earth and determination of the conditions favoring detachment on earth [14,16].
- Development of a new method for coating the interior of silica growth ampoules with transparent boron nitride, which yields the high contact angles for semiconductor melts that favor detachment [14,15].
- Development of a new coating for the interior of Pyrex ampoules yielding very high contact angles for water and molten organic compounds [13].
- Development of a material-balance model for steady detached solidification that provides greatly improved insight into the process, both in microgravity and on earth [9,10]. Dimensionless parameters were found that clarify the role of operating conditions and physical properties. The reason for the occurrence of two steady states was clarified, along with their relative stability.
- Observation of periodic gas tubes during directional solidification of water and organic compounds [9,11,13]. Gas bubbles did not propagate around the periphery to yield full detachment. Failure to obtain detachment is attributed to the ready plastic deformation of these materials, so that the frozen material continues to adhere to the ampoule wall during cooling.

## **Publications resulting from this grant**

1. L.L. Regel and W.R. Wilcox, "Detached Solidification," Proceedings of the First Pan Pacific Basin Workshop on Microgravity Sciences, J. Jap. Soc. Microgravity Appl. 15 (1998) 460-465.
2. L.L. Regel and W.R. Wilcox, "Improved Crystal Quality by Detached Solidification in Microgravity," Proceedings of the 1998 Microgravity Materials Science Conference, compiled by D.C. Gillies and D.E. McCauley, NASA/CP-1999-209092, Marshall Space Flight Center, pp 533-540 (1999).
3. Dmitri Popov, "Modeling of Detached Solidification and Unsteady Eutectic Solidification," Ph.D. Thesis, Clarkson University, 1999.
4. L.L. Regel and W.R. Wilcox, "Detached Solidification in Microgravity: A Review," Microgravity Sci. Technol. 14, 152-166 (1999). Here in Appendix A.
5. Y. Wang, L.L. Regel and W.R. Wilcox, "Influence of Contact Angle, Growth Angle and Melt Surface Tension on Detached Solidification of InSb," J. Crystal Growth 209, 175-180 (2000). Here in Appendix B.
6. Y. Wang, L.L. Regel and W.R. Wilcox, "Steady State Detached Solidification of Water at Zero Gravity," J. Crystal Growth 226, 430-435 (2001). Here in Appendix C.
7. L.L. Regel, W.R. Wilcox, Y. Wang and C. Burkhard, "Improved Crystal Quality by Detached Solidification in Microgravity," Proceedings of the Microgravity Materials Science Conference 2000, edited by N. Ramachandran, N. Bennett, D. McCauley, K. Murphy, S. Poindexter, NASA/CP-2001-210827, pp 487-492 (2001).
8. W.R. Wilcox and L.L. Regel, "Microgravity Effects on Materials Processing: A Review," Proceedings of the 7<sup>th</sup> European Conference on Advanced Materials and Processes, Associazione Italiana di Metallurgia, Milano (2001). Here in Appendix D.
9. Yazhen Wang, "Modeling and Ground-Based Experiments on Detached Solidification," Ph.D. Thesis, Clarkson University, 2001.
10. Y. Wang, L.L. Regel and W.R. Wilcox, "Approximate Material Balance Solution to the Moving Meniscus Model of Detached Solidification," J. Crystal Growth 243, 546-560 (2002). Here in Appendix E.
11. Y. Wang, L.L. Regel, and W.R. Wilcox, "Can propagation of gas bubbles lead to detached solidification? Experiments on freezing of water," Crystal Growth & Design 2, 453-461 (2002). Here in Appendix F.
12. L.L. Regel, W.R. Wilcox, Y. Wang and J. Wang, "Improved Crystal Quality by Detached Solidification in Microgravity," Proceedings of the 2002 NASA Microgravity Materials Science Conference (2002).
13. Craig Burkhard, "Solidification of Zone-Refined Naphthalene and High Contact-Angle Coatings," M.S. Thesis, Clarkson University, 2002.
14. Jianbin Wang, "Detached Solidification of Indium Antimonide in Pyrolytic Boron Nitride," M.S. Thesis, Clarkson University, 2002.
15. Jianbin Wang, Liya L. Regel and William R. Wilcox, "Inexpensive method for coating the interior of silica growth ampoules with pyrolytic boron nitride," submitted for publication in 2003. Appendix G.
16. Jianbin Wang, Liya L. Regel and William R. Wilcox, "Detached solidification of InSb on earth," submitted for publication in 2003. Appendix H.

## **Presentations**

- a. L.L. Regel and W.R. Wilcox, "Detached Solidification," First Pan-Pacific Basin Workshop, 4th Japan-China Workshop on Microgravity, Waseda (June 1998).
- b. L.L. Regel and W.R. Wilcox, "Improved Crystal Quality by Detached Solidification in Microgravity," 1998 Microgravity Materials Science Conference, Huntsville (June 1998).
- c. L.L. Regel, W.R. Wilcox, Y. Wang, D. Popov and C. Burkhard, "Experimental and Theoretical Modeling of Detached Solidification," American Conference on Crystal Growth and Epitaxy, Tucson (August 1999).
- d. C. Burkhard, L. Regel and W. Wilcox, "New non-wetting coatings," New York State Center for Advanced Materials Processing, Lake Placid (May 2000).
- e. Y. Wang, L. Regel and W. Wilcox, "Gas tubes and bubbles in Ice Cubes," New York State Center for Advanced Materials Processing, Lake Placid (May 2000).
- f. C. Burkhard, L.L. Regel and W.R. Wilcox, "New Non-Wetting Coatings for Detached Solidification Studies," Centrifugal Materials Processing IV, Potsdam (May 2000).
- g. L.L. Regel, W.R. Wilcox, Y. Wang and C. Burkhard, "Improved crystal quality by detached solidification in microgravity," Microgravity Materials Science Conference, Huntsville (June 2000).
- h. W.R. Wilcox and L.L. Regel, "Microgravity Effects on Materials Processing: A Review," 7<sup>th</sup> European Conference on Advanced Materials and Processes, Rimini, Italy (June 2001).
- i. L. Regel, Y. Wang, C. Burkhard and W. Wilcox, "Periodic Gas Tubes Formed during Directional Solidification," Thirteenth American Conference on Crystal Growth and Epitaxy (ACCGE-13), Burlington, Vermont (August 2001).
- j. W. Wilcox, Y. Wang and L. Regel, "Material Balance for Detached Solidification," ACCGE-13, Burlington (August 2001).
- k. Y. Wang, L.L. Regel and W.R. Wilcox, "Material Balance for Detached Solidification," International Astronautical Congress, Toulouse (October 2001).
- l. Y. Wang, C. Burkhard, L.L. Regel and W.R. Wilcox, "Periodic Gas Tubes formed during Directional Solidification," International Astronautical Congress, Toulouse (October 2001).
- m. W.R. Wilcox and L.L. Regel, "Detached Solidification," MAE Department, Clarkson University (November 2001).
- n. J.B. Wang, L.L. Regel and W.R. Wilcox, "Achievement of Detached Solidification on Earth," Annual Technical Meeting, New York State Center for Advanced Materials Processing, Saratoga Springs (May 2002).
- o. L.L. Regel, W.R. Wilcox, Y. Wang and J. Wang, "Improved Crystal Quality by Detached Solidification in Microgravity," NASA Microgravity Materials Science Conference, Huntsville (June 2002).